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WATER QUALITY BASIC DATA REPORT ON ANALYTICAL REFERENCE SAMPLE TYPE I, BD-1

Robert A. Taft Sanitary Engineering Center
Cincinnati, Ohio

4 April 1958

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Report on

ANALYTICAL REFERENCE SAMPLE

TYPE I, HD-1

April 4, 1958

U.S. Department of Health, Education, and Welfare
Public Health Service
Robert A. ~~Taft~~ Sanitary Engineering Center
Cincinnati, Ohio

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PARTICIPANTS IN STUDY OF SAMPLE TYPE I, HD-1
(Distributed January 6, 1958)

Board of Public Utilities
Kansas City, Kansas

Corps of Engineers
Dalecarlia Filter Plant
Washington, D. C.

Bismarck Water Department
Bismarck, North Dakota

City of St. Louis Water Department
St. Louis, Missouri

St. Louis County Water Company
University City, Missouri

Hagerstown Water Plant
Hagerstown, Maryland

Chemical Laboratory
Mississippi State Board of Health
Jackson, Mississippi

City Water Department
Buffalo, New York

Erie County Department of Health
Buffalo, New York

Huntington Water Corporation
Huntington, West Virginia

City Water Department
Pasco, Washington

Arizona Water Company
Yuma, Arizona

City Water Department
Torresdale Laboratory
Philadelphia, Pennsylvania

City Board of Water Commissioners
Detroit, Michigan

City Water Department
Williston, North Dakota

Metropolitan Utilities District
Omaha, Nebraska

City Water Department
Laredo, Texas

Minneapolis-St. Paul Sanitary District
St. Paul, Minnesota

St. Joseph Water Company
St. Joseph, Missouri

Water, Gas & Sewage Treatment Dept.
Duluth, Minnesota

City Water Department
Cincinnati, Ohio

City Water Department
Weiser, Idaho

INTRODUCTION

Since the initiation of the Water Quality Basic Data Program in October of 1957, analytical data are being developed and accumulated in a steadily increasing volume. Because these analyses originate from so many different laboratories, it is essential that they be comparable as to precision and accuracy.

The reference sample technique is a mechanism by which this objective can be achieved. Reference samples will be submitted to each of the collaborating laboratories at periodic intervals to assure continued precision and accuracy of the data submitted.

REPORT ON THE ANALYTICAL REFERENCE SERVICE SAMPLE TYPE I, BD-1

This report summarizes the results for Analytical Reference Service Sample Type I, B D-1, submitted to twenty laboratories cooperating in the Water Quality Basic Data Program of the Public Health Service. Two of the laboratories submitted their results too late for inclusion in this report.

This is the first in the series of standard samples which will be routinely submitted to the cooperating agencies. Through this means, the program is assured of the precision and accuracy of the data submitted. This report may also be used as a means of self-evaluation by each of the participating agencies. In cases where a laboratory obtained a result which was not in agreement with the known value (or varied by more than two standard deviations) a review of procedure or technique may be indicated.

The analyses requested on this sample were some of those which are commonly used in the chemical and physical characterization of water. Most laboratories generally have little or no difficulty in performing these examinations. The overall excellence of the data indicates that the large majority of laboratories are obtaining valid analytical results. Those laboratories which had reported errors beyond the acceptable limits have already been informed.

The individual results as reported by the various laboratories are presented in Table 1 and the bar graphs . The dotted lines on the bargraphs represent the limits of error in terms of two standard deviations for each particular test. Assistance on laboratory procedures is available to the cooperating laboratories from the Public Health Service, through the Regional Offices or the Sanitary Engineering Center.

It is strongly desired that all participating laboratories supply comments on the analytical methods they use. A complete log of pertinent details, difficulties, modifications, sources of error, and other factors would be extremely valuable. Such a compilation of comments could represent an exchange of ideas and opinions that can well become a major part of the program.

DISCUSSION OF RESULTS

For purposes of clarity, the average figures on temperature and ammonia are not statistically treated in this report. The statistical limit used is the standard deviation, calculated in the usual manner using all data submitted. The raw data were then resurveyed, and those results differing by more than twice the standard deviation were eliminated. This tends to eliminate those results which contain gross errors resulting from use of erroneous dilution factors, badly standardized solutions, and other reasons. Within this limitation, 68% of all groups should have results within plus or minus one standard deviation and 95% of all groups should be within plus or minus two standard deviations.

AMMONIA

The ammonia determination was requested with the foreknowledge that the constituent would not be stable. It was desirable, however, to determine how much change would be found by the individual laboratories. It was expected that the concentration of ammonia would decrease over a period of time, and no result should be in excess of the amount added. On the other hand there is no means of determining whether any reported result which was less than the amount originally added to the standard sample is actually in error. Because of these factors those laboratories reporting results greater than 0.30 mg/l should be alerted to possible errors in method.

CHLORIDE

Although several methods are available for this determination, the Mohr and mercuric nitrate procedures are most frequently used. The endpoint in each method is reasonably sharp and clear cut so that only highly colored samples or samples with unusual interferences are apt to cause difficulties. The chief precaution to be observed in this determination, as in any volumetric procedure, is standardization of the reagents.

On this measurement, performed by 16 of the 18 participating groups, only two laboratories submitted erroneous results, one of which was an error in calculation. The standard deviation of 3.0 mg/l indicated that on this sample, approximately 65% of the laboratories obtained results which did not deviate by more than this amount from the true concentration of 101 mg/l and that approximately 95% of all laboratories obtained values within 6.0 mg/l of the true amount.

SULFATE

The gravimetric sulfate determination is a straight-forward procedure with no serious inherent difficulties. On this particular sample it was necessary to remove all of the turbidity prior to sulfate precipitation. If this operation was not carefully performed, a high result could be obtained. At the same time the final precipitate had to be thoroughly dried to avoid another positive error. Only three laboratories reported figures which might indicate insufficient drying or faulty removal of turbidity. One laboratory submitted a result sufficiently below the true value to indicate incomplete precipitation or faulty precipitate recovery. Two groups submitted good results based on a turbidimetric method and one group measured sulfate using the tetrahydroxyquinone volumetric procedure with equally good results. In general, all laboratories performed this analysis exceptionally well, the standard deviation being only 7.3 mg/l at a concentration of 240 mg/l.

TOTAL DISSOLVED SOLIDS

In the performance of the Total Dissolved Solids determination no difficulties should normally be encountered. A measured volume of turbidity-free sample is merely placed in a dish, evaporated to dryness and weighed. Two precautions must be observed, however; the turbidity must be completely removed and the sample must be taken to dryness at 103-105°C. Incomplete drying allows water of hydration to be included in the final weighing which, along with turbidity particles, can cause a result in excess of the true amount. A third precaution, also related to drying, must be exercised. If the drying temperature is too high certain materials such as carbonates, bicarbonates, or chlorides may be volatilized and the resulting figure will be too low. For these reasons the precautions, while simple, must be strictly observed.

The standard deviation of 17.7 mg/l for this determination indicates that the analysis was properly performed by the majority of the laboratories. Comparing this standard deviation to that obtained on the sulfate measurement (17.7 mg/l vs. 7.3 mg/l) also indicates the necessity for attention to minor details in this method.

ALKALINITY

This is a volumetric procedure which allows a choice of indicators or an electrometric measurement for end point detection. Use of the methyl orange end point frequently leads to significant deviations in the titration. The reports do not indicate which methods were used for this measurement but the results by all laboratories are in good agreement. No laboratory reported a result containing a significant error. The standard deviation was only 2.6 mg/l at a concentration of 64 mg/l.

HARDNESS

For the hardness determination all laboratories except one employed some modification of the EDTA titration, the differences in technique being mainly related to preparation of the indicator. The titration in general presents no difficulty, the end point being easy to detect. The standard deviation of 8.2 mg/l with a concentration of 250 mg/l hardness (as $CaCO_3$) indicates that all groups using the EDTA titration performed very well.

TURBIDITY

The turbidity determination has many modifications, but the standard procedure is still the Jackson Turbidimeter. For this reason it was requested that all results be reported in Jackson units. If a laboratory chose to use a photometric instrument for the measurement, it was asked to convert from the photometer reading to a Jackson reading. The standard deviation based on the Jackson Turbidimeter was 19.4. Reported data ranged from 83 to 50. Using other instruments the standard deviation was 49.1, with a range of 100 to 2.

It was unfortunate that very few readings approached the value of 88, the quantity originally present in the prepared samples. The errors could have been due to one or more of the following reasons:

(1) The turbidity particles agglomerated, giving the effect of fewer but larger particles.

(2) The particles coated the walls of the sample bottle and were not removed with the sample.

(3) The particles settled and the sample was not sufficiently agitated before withdrawal of the aliquot.

(4) The inherent weaknesses of the method itself, such as steadiness of the flame, amount of light in the area, and judgment of the operator.

The first three reasons cited would give a low result and all of the results, with one exception, were lower than the known turbidity of the fresh sample.

HYDROGEN-ION CONCENTRATION, pH

The true pH of this sample is the resultant of the various chemicals added to the substrate to furnish the desired concentration of other ions.

The figure reported under amount added represents the result obtained in the Sanitary Engineering Center laboratory using a carefully standardized instrument, immediately after the sample was prepared.

It can be seen that the precision among the laboratories performing this measurement was very good. Sixteen groups used electrometric measurement while the remaining two used color standards. Not enough data are available on the color method to draw any conclusions, although it is generally agreed that the electrometric procedure is preferable. As was expected, the pH readings were somewhat lower than the initial values obtained at the time of sample preparation.

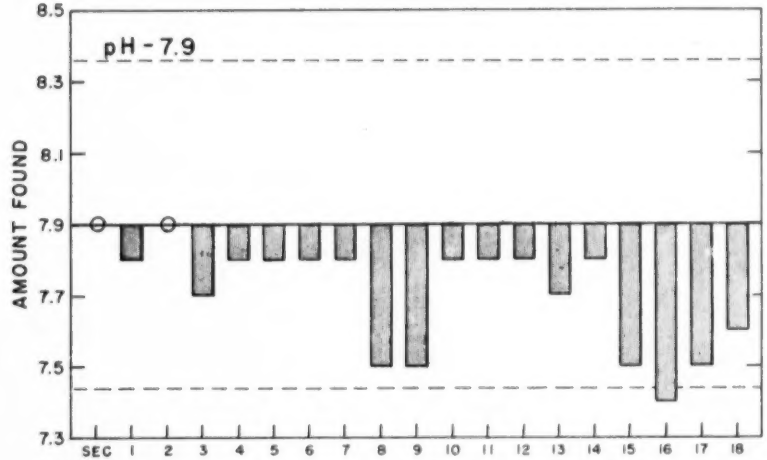
TABLE 1 - SUMMARY OF RESULTS, ANALYTICAL REFERENCE SERVICE SAMPLE TYPE I, ED-1.

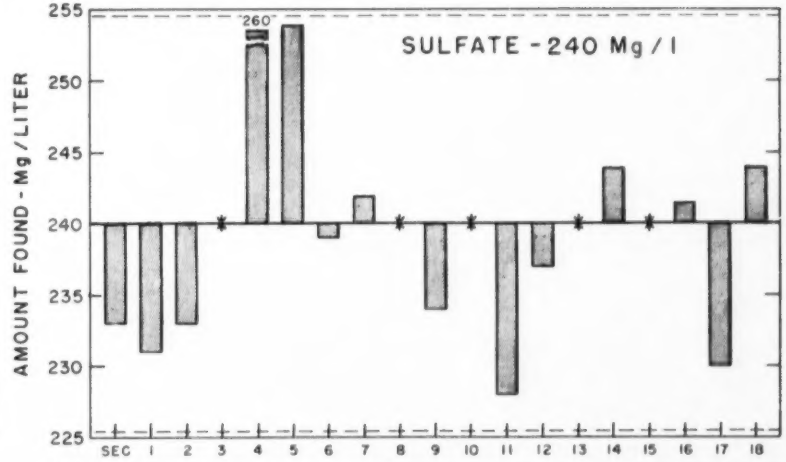
Analysis Performed	Chloride	Sulfate	Total Dissolved Solids	Alkalinity	Hardness	Turbidity	pH	NH_3
Amount Added	101 mg/l	240 mg/l	613 mg/l	64 mg/l	250 mg/l	88 Jackson Units	7.95 mg/l	0.30 mg/l
Laboratory No.	Average Amount Reported by All Laboratories							
1	100	231	544*	60	230	80 J. U.	7.8	.48
2	105	233	601	67	250	78	7.9	.23
3	185*	---	---	57*	241	71	7.7*	---
4	99	260*	640	66	244	69	7.8	.20
5	99	254	645	66	244	54	7.8	---
6	105	239	619	68	250	50	7.8	.17
7	103	242	605	64	248	70	7.8	.45
8	97	---	615	64	240	65	7.5	.12
9	105	234	578	62	245	40	7.5	.15
10	97	---	---	69	260	48	7.8	---
11	101	228	522*	62	248	83	7.8	0.6
12	97	237	605	61	242	76	7.8	.14
13	---	---	---	66	231	75	7.7	---
14	107	244	635	63	250	30	7.8	.08
15	---	---	610	60	240	2*	7.5	---
16	100	242	608	62	250	63	7.4	.24
17	120*	230	600	65	190*	100	7.5	---
18	103	244	---	65	250	67	7.6*	---
SEC	102	233	624	63	248	88	7.95	0.32
Arithmetic Mean	107	239	603	64	242	64	7.7	---
Median	102	238	608	64	245	69	7.8	---
Standard Deviation	3.0	7.3	17.7	2.6	8.2	28	---	---

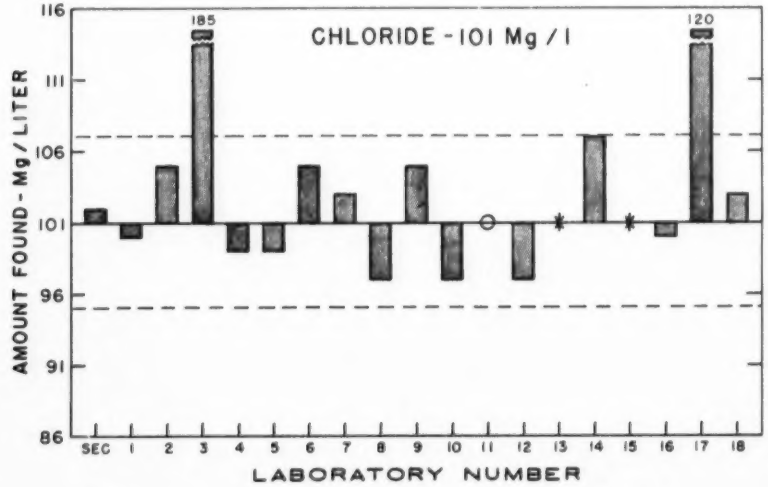
*Indicates datum not used in calculation of standard deviation.

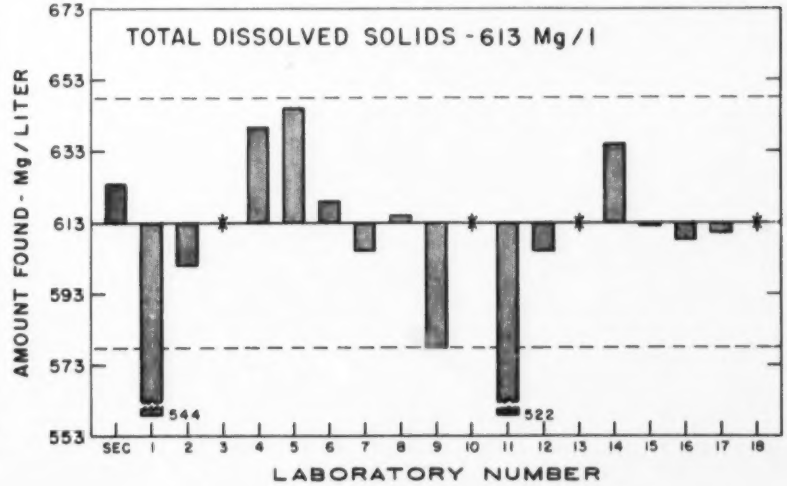
WATER QUALITY BASIC DATA

ANALYTICAL REFERENCE SERVICE
SAMPLE TYPE I, BD-I





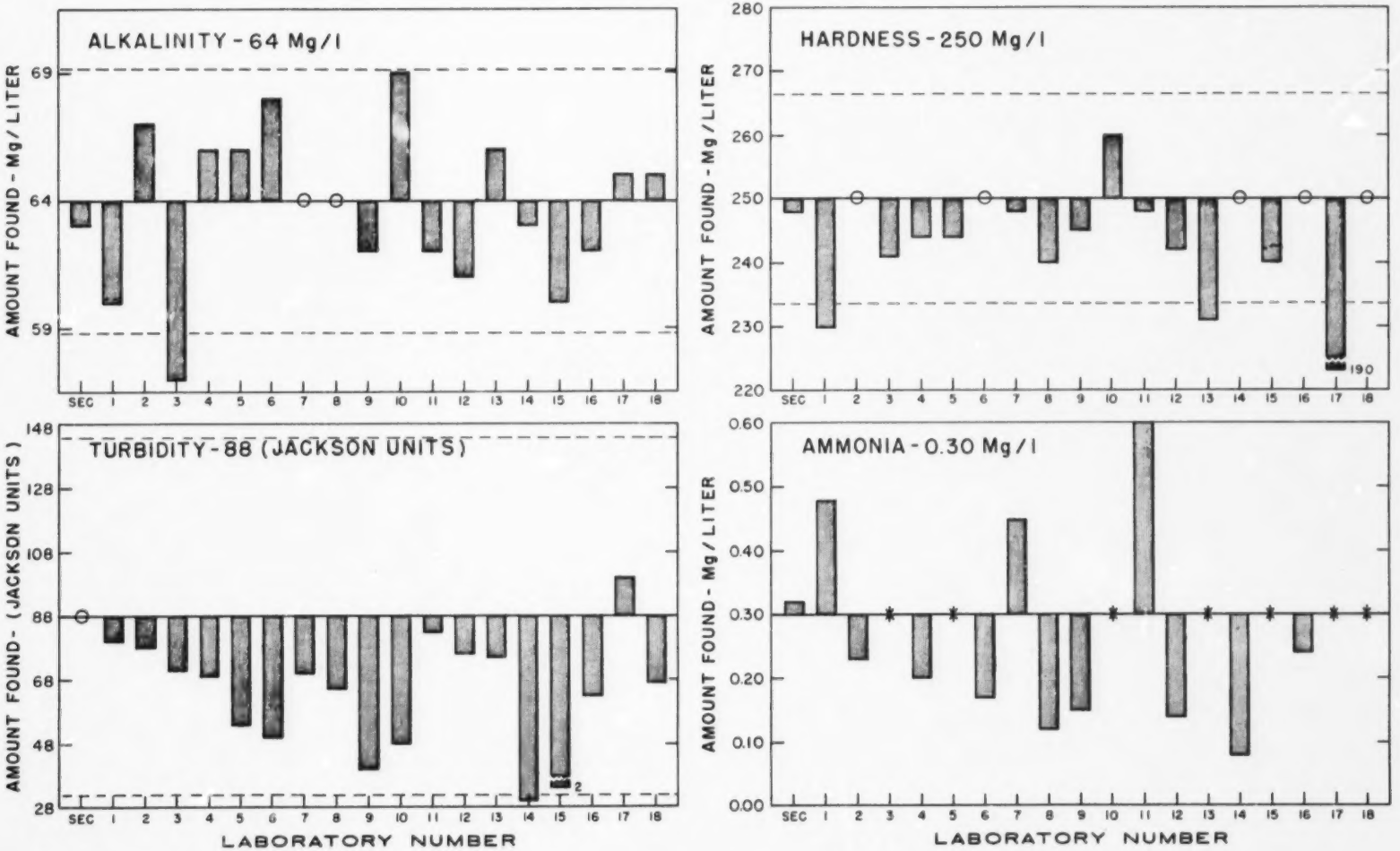




NOTE:
O = PERFECT RESULTS
* = NO ANALYSIS PERFORMED

WATER QUALITY BASIC DATA

ANALYTICAL REFERENCE SERVICE
SAMPLE TYPE I, BD-I



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